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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: Jiann H. Chen et al.)
Serial No.: 09/608,818)
Filed: June 30, 2000)
For: METHOD OF CURING A FUSER MEMBER)
OVERCOAT AT LOW TEMPERATURES)

#13
Examiner: *Freida*
E. Tsoy
1/27/03
Art Unit:
1762

APPEAL UNDER 37 CFR §1.191

Commissioner for Patents
Washington, D.C. 20231
Box AF

Having filed a Notice of Appeal on November 18, 2002, the appellants hereby submit this Appeal Brief in triplicate.

Real Party in Interest

The real party in interest is NexPress Solutions LLC, 1447 St. Paul Street, Rochester, New York 14653-7102.

Related Appeals and Interferences

There are no related appeals or interferences that will directly affect, or be directly affected by, or have a bearing on the Board's decision in this appeal.

Status of Claims

In an Office Action mailed July 17, 2002, all claims were finally rejected. Claims 1-20 were finally rejected under 35 U.S.C. §103(a) as being unpatentable over Hartley et al., U.S. Patent No. 4,853,737 ("Hartley") and, incorporated by reference, Lentz, U.S. Patent No. 4,257,699 ("Lentz") in view of Schlueter, Jr. et al., U.S. Patent No. 5,995,796 ("Schlueter"). Claims 1-22 were finally rejected under 35 U.S.C. §103(a) as being unpatentable over Hartley in view of Schlueter and Blong et al., U.S. Patent No. 5,527,858. ("Blong").

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Status of Amendments

In an Office Action mailed March 5, 2002, claims 4 and 10 were rejected under 35 U.S.C. 35 §112, second paragraph, for failing to particularly point out and distinctly claim the subject matter that the applicant regards as the invention. Also, claims 1-20 were rejected under 35 U.S.C. §103(a) as being unpatentable over Hartley and, incorporated by reference, Lentz in view of Schlueter.

In an Amendment filed May 24, 2002, claims 1, 4, and 10 were amended, and new claims 21 and 22 were added.

In an Office Action mailed July 17, 2002, the §112, second paragraph rejection of claims 4 and 10 was withdrawn, but the §103(a) rejection of claims 1-20 as unpatentable over Hartley, Lentz, and Schlueter was maintained and made final. In addition, claims 1-22 were finally rejected under 35 U.S.C. §103(a) as being unpatentable over Hartley, Schlueter, and Blong. These final rejections were reiterated in an Advisory Action mailed September 19, 2002.

Summary of the Invention

The present invention is directed to a method for making a fuser member in which a coating composition contained in an organic solvent is coated on the support, thereby forming a layer of the coating composition on the support. The coating composition comprises a fluorocarbon thermoplastic random copolymer, a curing agent having a bisphenol residue, a particulate filler containing zinc oxide, antimony-doped tin oxide particles, and an aminosiloxane; the fluorocarbon thermoplastic random copolymer has subunits of $-(CH_2CF_2)_x-$, $-(CF_2CF(CF_3)_y-$, and $-(CF_2CF_2)_z-$, wherein x is from 1 to 50 or 60 to 80 mole percent, y is from 10 to 90 mole percent, z is from 10 to 90 mole percent, and x + y + z equals 100 mole percent. The layer of the coating composition on the support is cured for 5 to 10 hours at a temperature in the range of 25°C to 120°C.

The claims read on the specification as follows:

| <u>Claim</u> | <u>Reference to Specification</u> |
|--------------|---|
| 1 | page 6, line 21 to page 7, line 8; page 13, line 12 to page 14, line 2 |
| 2 | page 7, line 9 |
| 3 | page 7, lines 9-12 |
| 4 | page 10, lines 7-9 |
| 5 | page 10, lines 7-9 |
| 6 | page 10, lines 7-9 |

7 page 10, lines 13-14 and 30-32
8 page 10, line 30 to page 11, line 5
9 page 12, lines 10-15 and 18-19
10 page 8, lines 11-13
11 page 12, lines 11-14
12 page 9, lines 13-18
13 page 9, lines 18-19
14 page 9, line 19
15 page 10, lines 14-15; page 11, lines 10-17;
 page 15, lines 3-19
16 page 11, lines 24-27
17 page 7, line 13
18 page 7, lines 13-15
19 page 10, lines 15-16
20 page 7, lines 13-14
21 page 10, lines 23-26
22 page 18, Table 2

Issues

As already noted, the appellants' invention is directed to a method for making a fuser member in which a coating composition comprising a fluorocarbon thermoplastic random copolymer, a curing agent, a zinc oxide filler, antimony-doped tin oxide particles, and an aminosiloxane is coated on a support, forming a layer that is cured at a low temperature of 25-125°C, preferably, 25-50°C, more preferably, 25°C. In the final §103(a) rejection of all the claims, the primary cited reference, Hartley, as well as two of the secondary references, Lentz and Schlueter, teach fuser roll release layers formed from fluoroelastomers that are cured at substantially higher temperatures. The issue to be resolved is the propriety of the rejections of the appellants' claims as being unpatentable over the disclosures of the cited references.

Grouping of Claims

All of the pending claims, 1-22, stand or fall together.

Argument

Fluorocarbon elastomers and fluorocarbon thermoplastics are well recognized as distinctly different materials characterized by substantially different physical properties, for example, surface energies (high for fluoroelastomers, low for fluoroplastics), glass transition temperatures (applicable to fluoroelastomers), and melting ranges (applicable to fluoroplastics). As discussed in Encyclopedia of

Chemical Technology, Fourth Edition, 1993, Volume 8, page 990 , a copy of which is enclosed as Attachment A, "Fluorocarbon elastomers are synthetic, noncrystalline polymers that exhibit elastomeric properties when cross-linked....In the 1960s, terpolymers of vinylidene fluoride, hexafluoropropylene, and tetrafluoroethylene, CF₂=CF₂, were developed and were commercialized by DuPont as Viton B." (emphasis added)

Attachment B, entitled "Comparison of Dupont Dow Viton® Fluoroelastomers," includes a description of Viton A as a fluoroelastomer dipolymer formed from vinylidene fluoride (VF₂) and hexafluoropropylene (HFP) and Viton B as a fluoroelastomer terpolymer formed from vinylidene fluoride (VF₂), hexafluoropropylene (HFP) and tetrafluoroethylene (TFE).

As described in Hull et al., "THV Fluoroplastic" in *Modern Fluoropolymers*, 1997, Chapter 13, pages 257-259, a copy of which is enclosed as Attachment C, a melt-processable fluoroplastic consisting of tetrafluoroethylene (TFE), hexafluoropropylene (HFP), and vinylidene fluoride (VF₂, as referred to as VDF) was developed by Hoechst AG in the early 1980s and is now marketed as Dyneon™ THV fluorothermoplastic. As mentioned on page 259, THV fluorothermoplastic is typically processed by melt extrusion at temperatures in the range of 230-250°C. It should be noted that THV fluorothermoplastic is formed from the same three monomers as the fluoroelastomer Viton B.

Attachment D, entitled "Properties of Fluoroplastics," lists the material properties for two THV fluorothermoplastics, THV-400 and THV-500, which differ from one another primarily in their melting temperatures and specific gravities.

Hartley, the primary reference cited in the rejection of the claims of the instant application, discloses a fuser roll having an outer layer that comprises cured fluorooelastomer having pendant polydiorganosiloxane segments that are covalently bonded to the backbone of the fluoroelastomer and have a number average molecular weight in the range of about 1,000 to 20,000.

Hartley teaches a fluoroelastomer base polymer such as Viton A or Viton B (column 2, line 29, to column 3, line 4), to which is covalently bonded a small amount of polydiorganosiloxane segments (column 4, line 40, to column 5, line 66), thereby providing an "internal lubricant" that results in a relatively low surface energy coating (column 4, lines 40-52). Hartley repeatedly and consistently refers to the disclosed compositions as fluoroelastomers and further frequently refers to the fluoroelastomer

"backbone" (for example, column 3, lines 24-29; column 4, line 60; column 8, lines 25-26). Incidentally, the compositions described in the illustrative examples of Hartley all contain the VF-HFP copolymer Viton A, along with the amino-terminated polydimethylsiloxane oligomer.

In the September 19, 2002 Advisory Action, despite these consistent, repeated teachings of Hartley, the Examiner asserted that the disclosed fluoroelastomers of Hartley are, in fact, fluorocarbon thermoplastic random copolymers, as claimed in the instant application, and further asserted that the pendant polydiorganosiloxane are covalently bonded to a thermoplastic VF-HFP-PTFE terpolymer backbone.

Hartley, in contrast to the instant specification, does not recite percentage ranges for the monomers comprising the disclosed fluoroelastomers, i.e., the VF-HFP copolymer Viton A, and the VF-HFP-TFE terpolymer Viton B. In the March 5, 2002 Office Action, the Examiner, citing the disclosure of Eddy et al., U.S. Patent No. 5,017,432 ("Eddy") at column 6, lines 4-6, that the terpolymer Viton B contains 61% VF, 17% HFP, and 22% TFE, alleges that, because these amounts fall within the monomer ranges recited for the thermoplastic fluoropolymers in the instant specification, the fluoropolymers of Hartley, specifically Viton B, must also be thermoplastic. However Eddy, at column 2, line 66 to column 3, line 6, and column 3, lines 17-26, clearly refers to Viton polymers, including Viton B, as elastomers, consistent with the characterization of these materials in the *Encyclopedia of Chemical Technology* article as well as by their commercial suppliers, DuPont and Dow.

As already noted, both the fluoroelastomer Viton B and THV fluorothermoplastics are formed from the same three monomers. Although the monomeric compositions of both of these commercial products may fall within the ranges recited in instant claim 1, it is well understood in the art that fluoroelastomers and fluorothermoplastics are distinctly different materials. For example, Effenberger et al., U.S. Patent No. 5,194,335 ("Effenberger"), cited in the July 17, 2002 Office Action, teaches a fluoroplastic resin dispersion modified by the addition of a fluoroelastomer latex (Abstract, emphasis added). On page 10 of that Office Action, the Examiner misstates that a THV fluoroelastomer is added to PTFE or PEF. THV is not a fluoroelastomer but, as described in the "THV Fluoroplastic" article, a fluoroplastic with improved properties compared with previously known fluoroplastics such as PTFE. At column 4, lines 6-47, Effenberger discloses suitable

fluoroelastomers for mixing with a fluoroplastic and mentions Viton materials among others. THV is not included in this listing because it is, in fact, not a fluoroelastomer but rather a fluoroplastic.

In further support of her assertion that the fluoropolymers of Hartley are thermoplastic polymers, the Examiner in the September 19, 2002 Advisory Action cited Shifman et al., U.S. Patent No. 6,203,873 ("Shifman"), which discloses a blend of a first fluorointerpolymer having elastomeric characteristics and a second fluorointerpolymer having thermoplastic characteristics (column 1, lines 46-48). Shifman describes the barrier layer 10 of the invention as being formed from a blend of at least two fluorointerpolymers, wherein at least one of the fluorointerpolymers is characterized as a fluoroelastomer and at least one of the fluorointerpolymers is characterized as a fluoroplastic (column 4, lines 33-37)..

Schifman further discloses that the first fluorointerpolymer having elastomeric characteristics and the second fluorointerpolymer having thermoplastic characteristics each comprises a copolymer, terpolymer, or mixture thereof formed by the copolymerization of two or more monomers selected from the group consisting of HFP, VF, and TFE (column 1, line 57 to column 2, line 5). Thus, both the elastomeric and the thermoplastic fluorointerpolymers taught by Schifman are formed from the same group of three monomers that also constitute the fluorocarbon thermoplastic random copolymer included in the coating composition of the present invention.

The appellants therefore respectfully reiterate that their fuser roll release layer is formed from a coating composition containing a fluorocarbon thermoplastic random copolymer, in contrast to Hartley, Lentz, and Schlueter, all of which teach fuser roll release layers formed from cured fluoroelastomers.

The Examiner has acknowledged that Hartley fails to teach the inclusion in the composition of antimony doped tin oxide, relying on Schlueter to supply this missing disclosure. As taught at page 11, line 28 to page 12, line 3 of the instant specification, the inclusion of antimony doped tin oxide particles is crucial for drastically lowering the curing temperature of the coated thermoplastic polymer, from 220 to 280°C to as low as room temperature (25°C). The elastomeric compositions of Schlueter include antimony doped tin oxide as an electrically conductive filler, but the reference cites no curing temperature, simply stating that the composition is subjected to a "step heat cure" for approximately 24 hours. Hartley teaches curing temperatures of at least 230°C, and Lentz includes an example with a curing temperature of 232°C. There is

no teaching in any of the cited references of a metal oxide or combination of oxides being used to enable low temperature curing of a fluoropolymer layer.

As taught in the instant specification, high temperature curing of a fluoroelastomer release layer can cause damage to a fuser roll, for example, depolymerization of silicone rubber in the cushion layer. The method of the present invention advantageously provides for the curing of a layer formed from a fluorocarbon thermoplastic copolymer composition at a substantially lower temperature of 25°C to 120°C, preferably 25°C to 50°C, more preferably 25°C. This benefit is enabled by the inclusion of antimony-doped tin oxide particles in the coating composition.

The compositions disclosed in Blong, in contrast to those of Hartley, Lentz and Schlueter, do include thermoplastic fluoropolymers. At column 1, lines 11-23, Blong refers to a relatively new class of commercially available fluoropolymers, which are thermoplastic terpolymers of TFE, HFP, and VF and are sold by the 3M Company as "3M THV fluoroplastics." In the illustrative examples in Blong, 3M THV 500 Fluoroplastic is employed (column 6, lines 33-37). The data sheets for THV-400 and THV-500 fluoroplastics enclosed as Attachment D shows that these materials have high melting temperatures and are typically processed by extrusion at even higher temperatures.

As disclosed at page 12, line 25 to page 13, line 2 of the instant specification, the same materials that are disclosed in Blong as melt-processable thermoplastic fluoropolymer components are employed in the coating compositions of the present invention, which can be cured at low temperatures. Blong, however, makes no mention of curing the disclosed compositions, reciting only an extrusion temperature of 230°C. This is not surprising, as fluoroplastics are commonly melt-processed by extrusion, with no included curing step.

It has already been noted, that the coating composition of the present invention comprises a fluorocarbon thermoplastic random copolymer having subunits of —(CH₂CF₂)_x—, —(CF₂CF(CF₃)_y—, and —(CF₂CF₂)_z—, wherein x is from 1 to 50 or 60 to 80 mole percent, y is from 10 to 90 mole percent, z is from 10 to 90 mole percent, and x + y + z equals 100 mole percent. This same fluorocarbon thermoplastic random copolymer having the same ranges of the three recited fluoromonomers is disclosed in the following issued patents:

U.S. 6,444,741
U.S. 6,429,249
U.S. 6,419,615
U.S. 6,416,819
U.S. 6,372,833
U.S. 6,361,829
U.S. 6,355,352

Hartley et al., U.S. 4,857,737 ("Hartley") is of record in all of these applications except U.S. 6,419,615.

Conclusion

The foregoing discussion makes clear that fluoroelastomers and fluorocarbon thermoplastics are well recognized in the art as distinct types of materials, even if they are formed from the same monomers in amounts falling within the same specified ranges. In the process of the present invention, a composition that includes a fluorocarbon thermoplastic random copolymer, a curing agent, and antimony-doped tin oxide particles is cured at low temperatures, in the range of 25°C to 120°C. The disclosures of Hartley, Lentz, and Schlueter all teach compositions comprising fluoroelastomers, which typically are cured at elevated temperatures. Blong, which does disclose a composition containing thermoplastic fluoropolymers, is silent with respect to curing of these polymers or to the inclusion of antimony-doped tin oxide particles in the composition. The teachings of these references are not combinable and, in any event, fail to render obvious the appellants' invention. Withdrawal of the §103(a) final rejection of claims 1-22 and allowance of this case is therefore earnestly solicited.

Respectfully submitted,

January 17, 2003
Date

Lee J. Fleckenstein
Lee J. Fleckenstein
Registration No. 36,136

JAECKLE FLEISCHMANN & MUGEL, LLP
39 State Street
Rochester, New York 14614-1310
Telephone: (585) 262-3640
Facsimile: (585) 262-4133



Appendix

1. A method of making a fuser member having a support comprising the steps of:

A) providing a support;

B) coating a coating composition contained in an organic solvent onto the support, thereby forming a layer of the coating composition on said support, said coating composition comprising a fluorocarbon thermoplastic random copolymer, a curing agent having a bisphenol residue, a particulate filler containing zinc oxide, antimony-doped tin oxide particles, and an aminosiloxane, the fluorocarbon thermoplastic random copolymer having subunits of:

$-(CH_2CF_2)_x-$, $-(CF_2CF(CF_3)_y-$, and $-(CF_2CF_2)_z-$,

wherein

x is from 1 to 50 or 60 to 80 mole percent,

y is from 10 to 90 mole percent,

z is from 10 to 90 mole percent,

x + y + z equals 100 mole percent; and

C) curing said layer of the coating composition on said support for 5 to 10 hours at a temperature in the range of 25°C to 120°C.

2. The method of claim 1 wherein the aminosiloxane is an amino functional polydimethyl siloxane copolymer.

3. The method of claim 2 wherein the amino functional polydimethyl siloxane copolymer comprises amino functional units selected from the group consisting of (aminoethylaminopropyl) methyl, (aminopropyl) methyl and (aminopropyl) dimethyl.

4. The method of claim 1 wherein the aminosiloxane has a total concentration in the coating composition of from 1 to 20 parts by weight per 100 parts of the fluorocarbon thermoplastic random copolymer.

5. The method of claim 1 wherein the aminosiloxane has a total concentration in the layer of from 5 to 15 parts by weight per 100 parts of the fluorocarbon thermoplastic random copolymer.

6. The method of claim 1 wherein the aminosiloxane has a total concentration in the layer of from 10 to 15 parts by weight per 100 parts of the fluorocarbon thermoplastic random copolymer.

7. The method of claim 1 wherein the zinc oxide has a total concentration in the layer of from 1 to 20 parts by weight per 100 parts of the fluorocarbon thermoplastic random copolymer.

8. The method of claim 1 wherein the zinc oxide has a total concentration in the layer of from 3 to 15 parts by weight per 100 parts of the fluorocarbon thermoplastic random copolymer.

9. The method of claim 2 wherein the fluorocarbon thermoplastic random copolymer is cured by bisphenol residues.

10. The method of claim 1 further comprising:
forming a cushion layer between said substrate and said layer of the coating composition.

11. The method of claim 1 wherein the fluorocarbon thermoplastic random copolymer is nucleophilic addition cured.

12. The method of claim 1 wherein x is from 30 to 50 mole percent, y is from 10 to 90 mole percent, and z is from 10 to 90 mole percent.

13. The method of claim 1 wherein x is from 40 to 50 mole percent and y is from 10 to 15 mole percent.

14. The method of claim 1 wherein z is greater than 40 mole percent.

15. The method of claim 1 wherein the antimony-doped tin oxide particles have a total concentration of from 3 to 20 parts by weight per 100 parts of the fluorocarbon thermoplastic random copolymer.

16. The method of claim 1 wherein the antimony-doped tin oxide particles comprise 3 to 10 weight percent antimony.

17. The method of claim 1 wherein the fluorocarbon thermoplastic random copolymer further comprises a fluorinated resin.

18. The method of claim 17 wherein the fluorinated resin has a number average molecular weight of between 50,000 to 50,000,000.

19. The method of claim 17 wherein the ratio of fluorocarbon thermoplastic random copolymer to fluorinated resin is between 1 : 1 to 50 :1.

20. The method of claim 17 wherein the fluorinated resin is polytetrafluoroethylene or fluoroethylenepropylene.

21. The method of claim 1 wherein said temperature in step C) is in the range of 25°C to 50°C.

22. The method of claim 21 wherein said temperature in step C) is 25°C.

FLUOROCARBON ELASTOMERS

Fluorocarbon elastomers are synthetic, noncrystalline polymers that exhibit elastomeric properties when cross-linked. They are designed for demanding service applications in hostile environments characterized by broad temperature ranges and/or contact with chemicals, oils, or fuels.

Military interest in the development of fuel and thermal resistant elastomers for low temperature service created a need for fluorinated elastomers. In the early 1950s, the M. W. Kellogg Co. in a joint project with the U.S. Army Quartermaster Corps, and 3M in a joint project with the U.S. Air Force, developed two commercial fluorocarbon elastomers. The copolymers of vinylidene fluoride, $\text{CF}_2=\text{CH}_2$, and chlorotrifluoroethylene, $\text{CF}_2=\text{CFCl}$, became available from Kellogg in 1955 under the trademark of Kel-F (1-3) (see FLUORINE COMPOUNDS, ORGANIC—POLYCHLOROTRIFLUOROETHYLENE; POLY(VINYLDENE) FLUORIDES). In 1956, 3M introduced a polymer based on poly(1,1-dihydroperfluorobutyl acrylate) trademarked 3M Brand Fluororubber 1F4 (4). The poor balance of acid, steam, and heat resistance of the latter elastomer limited its commercial use.

In the late 1950s, the copolymers of vinylidene fluoride and hexafluoropropylene, $\text{CF}_2=\text{CFCF}_3$, were developed on a commercial scale by 3M (Fluorel) and by Du Pont (Viton) (5-8). In the 1960s, terpolymers of vinylidene fluoride, hexafluoropropylene, and tetrafluoroethylene, $\text{CF}_2=\text{CF}_2$, were developed (9) and were commercialized by Du Pont as Viton B. At about the same time, Montedison developed copolymers of vinylidene fluoride and 1-hydropentafluoropropylene as well as terpolymers of these monomers with tetrafluoroethylene, marketed as Tecnoflon polymers (10,11).

In the 1960s and 1970s, additional elastomers were developed by Du Pont under the Viton and Kalrez trademarks for improved low temperature and chemical resistance properties using perfluoro(methyl vinyl ether), $\text{CF}_2=\text{CFOCF}_3$, as a comonomer with vinylidene fluoride and/or tetrafluoroethylene (12,13) (see FLUORINE COMPOUNDS, ORGANIC—TETRAFLUOROETHYLENE POLYMERS AND COPOLYMERS).

Bromine- and iodine-containing fluoroolefins have been copolymerized with the above monomers in order to allow peroxide cure (14-21). The peroxide cure system does not require dehydrofluorination of the polymer backbone, resulting in an elastomer that shows improved properties after heat and fluid aging.

Copolymers of propylene and tetrafluoroethylene, which are sold under the Aflas trademark by 3M, have been added to the fluorocarbon elastomer family (21-26). Also 3M has introduced an incorporated cure copolymer of vinylidene fluoride, tetrafluoroethylene and propylene under the trademark Fluorel II (27). These two polymers (Aflas and Fluorel II) do not contain hexafluoropropylene. The substitution of hexafluoropropylene with propylene is the main reason why these polymers show excellent resistance toward high pH environments (28). Table 1 lists the principal commercial fluorocarbon elastomers in 1993.

Properties

Table 2 summarizes general characteristics of vulcanizates prepared from commercially available fluorocarbon elastomer gumstocks.



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Comparison of Dupont Dow Viton® Fluoroelastomers

There are three major general use grades of Viton® fluoroelastomer: A, B and F. They differ primarily in their resistance to fluids, and in particular aggressive lubricating oils and oxygenated fuels, such as methanol and ethanol automotive fuel blends. There is also a class of high performance Viton® grades: GB, GBL, GP, GLT, and GFLT.

General Use Grades:

Viton® A: (vinylidene fluoride and hexafluoropropylene)

Viton® A is a family of fluoroelastomer dipolymers, that is they are polymerized from two monomers, vinylidene fluoride (VF2) and hexafluoropropylene (HFP). Viton® A fluoroelastomers are general purpose types that are suited for general molded goods such as o-rings and v-rings, gaskets, and other simple and complex shapes. There is a full range of Viton® A grades that accomodate various manufacturing processes including transfer and injection molding, extrusion, compression molding, and calendering.

Viton® B: (vinylidene, hexafluoropropylene and tetrafluoroethylene)

Viton® B is a grade of fluoroelastomer terpolymers, that is they are polymerized from three monomers, vinylidene (VF2), hexafluoropropylene (HFP), and tetrafluoroethylene (TFE). Viton® B

fluoroelastomers offer better fluid resistance than A type fluoroelastomer. There is a full range of Viton® B grades that accommodate a variety of manufacturing processes including injection and compression molding, extrusion, and calendering.

Viton® F:

Viton® F is a grade of fluoroelastomer terpolymers, that is they are polymerized from three monomers, vinyl fluoride (VF2), hexafluoropropylene (HFP), and tetrafluoroethylene (TFE). Viton® F fluoroelastomers offer the best fluid resistance of all Viton® types. F types are particularly useful in applications requiring resistance to fuel permeation. There is a range of Viton® F grades to accommodate various manufacturing requirements.

High Performance Grade:

Viton® GB, GBL:

Viton® GB and GBL are grades of fluoroelastomer terpolymers, that is they are polymerized from three monomers, vinyl fluoride (VF2), hexafluoropropylene (HFP), and tetrafluoroethylene (TFE). Viton® GB and GBL use peroxide cure chemistry that result in superior resistance to steam, acid, and aggressive engine oils. There is full range of GB and GBL types that can accommodate most rubber processing requirements including compression, injection and transfer molding, extrusion, and calendering.

Viton® GLT:

Viton® GLT is a fluoroelastomer designed to retain the high heat and the chemical resistance of general use grades of Viton® fluoroelastomer, while improving the low temperature flexibility of the material. Glass transition temperatures (Tg) of materials are indicative of low temperature performance in typical elastomer applications. Viton® GLT shows an 8 to 12°C lower Tg than general use Viton® grades. There is a range of GLT products to accommodate various processing conditions.

13

THV Fluoroplastic

D. E. HULL, B. V. JOHNSON, I. P. RODRICKS and
J. B. STALEY
Fluothermoplastic Business Unit, Dyneon, St Paul, MN, USA

1 BACKGROUND

In the early 1980s Hoechst AG developed a commercial production process for a unique melt processable fluoroplastic consisting of tetrafluoroethylene, hexafluoropropylene, and vinylidene fluoride (Figure 13.1). The impetus for this product was a contract to develop an outdoor fabric coating that could provide the protection of typical fluoroplastics like PTFE and ETFE, but additionally could be used with PVC-coated polyester fabric without significantly compromising overall flexibility. PTFE and ETFE could not be used since their high melt fusion temperatures destroyed PVC-coated polyester fabric. Hoechst developed the product and conducted a restricted test market (primarily fabric coating within Germany) under the brandname HostaflonTM TFB X. In January 1993 Hoechst granted worldwide marketing rights to 3M and the product reached full commercial status under the name 3MTM THV Fluoroplastic. With the start-up of the Dyneon 3M/Hoechst joint venture in August 1996, the product is now known as DyneonTM THV fluorothermoplastic.

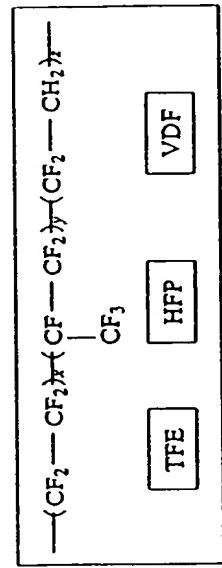


Figure 13.1. Chemical structure of THV terpolymer. (Reproduced with permission)

Attachment C

2 MANUFACTURING

THV Fluoroplastic is produced in Gendorf, Germany. THV Fluoroplastic is polymerized under aqueous emulsion conditions and the resulting dispersions can be sold and used directly or following concentration with an emulsifier. Dry forms result from coagulation, washing, filtering and drying of the dispersion followed by extrusion (pellets) or grinding (powder) operations. Additives are generally not added to THV Fluoroplastic since the product is inherently very stable and easy to process. An electrostatic dissipative compound has been developed and was commercialized in 1995.

Products are available in 25 kilogram bags or larger containers (pellets), 50 kilogram drums (powders) or 35 kilogram (wet weight dispersions) barrels. No chlorofluorocarbons are employed at any step during the production process and the relatively recent construction of the production facilities allowed implementation of state of the art technology.

3 PROPERTIES

In general, THV Fluoroplastic is set apart from other melt-processable fluoroplastics by a combination of properties that include relatively low processing temperatures; bondability (to itself and other substrates); high flexibility; excellent clarity and low refractive index; and efficient E-beam crosslinking. One grade, THV-200, is soluble in common organic solvents. THV Fluoroplastic also retains properties common to other commercial fluoroplastics such as chemical resistance, weatherability, and low flammability.

There are four commercial THV Fluoroplastic grades (three dry and one aqueous dispersion) differing in the monomer ratios that subsequently influence melting points, chemical resistance and flexibility. Table 13.1 shows the basic properties of the three dry grades. THV-200 has the lowest melting point, the least chemical resistance of the THV Fluoroplastic grades (soluble in common solvents such as ethyl acetate, ketones, etc.), is the easiest to E-beam crosslink and is the most flexible. THV-500 has the best chemical and permeation resistance of the THV Fluoroplastic grades. THV-400 has a slightly lower melting point than THV-500 to meet specific processing requirements.

THV Fluoroplastic is available in pellet (THV-200G, 400G and 500G); powder (THV-200P) and aqueous dispersions (THV-330R 30% solids and 350C 50% solids).

THV Fluoroplastic is processed within the fluoroplastics fabricator base, but because of its relatively low processing temperature it is also processed by olefinic processors. Virtually all of the melt-processable plastic processing methods are used with THV Fluoroplastic including extrusion, co-extrusion and

Table 13.1. Typical THV properties (nominal values, not for specification purposes). (Reproduced with permission)

| Property | ASTM method | THV grade | | |
|---|-------------------|-----------|---------|---------|
| | | THV 200 | THV 400 | THV 500 |
| Specific gravity | D792 | 1.95 | 1.97 | 1.98 |
| Melting range (°C) | D3418 | 115-125 | 150-160 | 165-180 |
| Thermal decomposition in air (°C) | TGA | 420 | 430 | 440 |
| Limiting oxygen index (LOI) | D2863 | 65 | NA | 75 |
| Tensile strength at break (psi) | D638 ^a | 4200 | 4100 | 4100 |
| Tensile strength at break (MPa) | D638 ^a | 29.0 | 28.3 | 28.3 |
| Elongation at break | D638 ^a | 600% | 500% | 500% |
| Flexural modulus (psi) | D790 | 12,000 | NA | 30,000 |
| Flexural modulus (MPa) | D790 | 82.7 | NA | 206.7 |
| Hardness, Shore D | D2240 | 44 | 53 | 54 |
| Dielectric constant at 23 °C | | | | |
| 100 kHz | D149 | 6.6 | 5.9 | 5.6 |
| 10 MHz | D149 | 4.6 | 4.1 | 3.9 |
| Melt flow index (gm/10 min @ 260 °C 5 kg) | D1238 | 20 | 10 | 10 |
| E-beam cured high temperature Resistance (°C) | NA | >150 | NA | NA |

^aProperty measured on extruded film

tandem extrusion; blow and coblow molding (including blown film); injection molding, skived film; vacuum forming; and solvent casting (only for THV-200). These processes produce film, tubing, containers, profiles and molded shapes. Many of these products involve multilayer constructions where THV Fluoroplastic provides chemical, barrier or other properties in a relatively thin layer bonded to thicker layers of structural plastics or elastomers of various types.

3.1 THV FLUOROPLASTIC DISTINGUISHING FEATURES

As mentioned earlier, THV Fluoroplastic has a unique combination of properties that set it apart from other melt-processable fluoroplastics. These features are beneficial not only to end-users but to manufacturers faced with part production and design challenges.

3.1.1 THV Fluoroplastic Processing Temperatures

During most extrusion processing, THV Fluoroplastic melt temperature at the die is in the 230-250 °C range. This relatively low processing temperature presents many new options for co-processing (e.g., coextrusion, cross-head extrusion, co-blow molding) with plastics as well as with various elastomers.

Attachment D

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State: THV-400

Material Properties**Mechanical Properties****Conditions**State 1
at break

State 2

| | | |
|-------------------------|-----|-------|
| Tensile Strength (MPa) | 23 | |
| Elongation at break (%) | 500 | |
| Hardness | 53 | Shore |

Physical & Electrical Properties**Cond**

State

| | |
|------------------|------|
| Specific Gravity | 1.97 |
|------------------|------|

Processing Properties**Conditions**

| | | |
|-----------------------------|-----|----------------------------------|
| Melting Temperature (°C) | 150 | Type |
| Processing Temperature (°C) | 243 | T_m , crystalline extrusion |

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Fluoroplastics

State: THV-500

Material Properties

Mechanical Properties

| | State 1 | State 2 |
|-------------------------|---------|----------|
| Flexural Modulus (MPa) | 207 | 23 °C |
| Tensile Strength (MPa) | 23 | at break |
| Elongation at break (%) | 500 | |
| Hardness | 54 | Shore |

Physical & Electrical Properties

| | Cond |
|------------------|-------|
| Specific Gravity | State |

1.78

Processing Properties

| | Conditions |
|--------------------------|------------|
| Melting Temperature (°C) | Type |

| | | |
|-----------------------------|-----|---------------------|
| Melting Temperature (°C) | 180 | T_m , crystalline |
| Processing Temperature (°C) | 249 | extrusion |

Suppliers

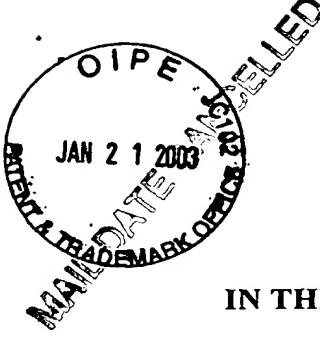
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| | |) | E. Tsoy |
| Serial No.: | 09/608,818 |) | |
| | |) | |
| Filed: | June 30, 2000 |) | Art Unit: |
| | |) | 1762 |
| For: | METHOD OF CURING A FUSER MEMBER OVERCOAT AT LOW TEMPERATURES |) | |
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